William E. Neff,
Fred Eller,
Kathleen Warner

National Center for
Agricultural Utilization
Research, Department
of Agriculture, Peoria,
USA

Composition of oils extracted from potato chips by supercritical fluid extraction*

To determine effects of two extraction procedures on oil compositions, tocopherols, monoacylglycerol, diacylglycerol, triacylglycerol, free fatty acids, polymers and polar components were determined in oils after extraction from potato chips by either supercritical carbon dioxide or hexane. Potato chips were fried in cottonseed oil or low linolenic acid soybean oil and sampled after 1, 10 and 20 h of oil use. Both extraction methods recovered comparable amounts of oil from the potato chips. Compositions of triacylglycerol and non-triacylglycerol components including tocopherols, monomer, polymer, monoacylglycerol, diacylglycerol were similar for samples of chips fried in either oil except for the δ-tocopherol data for potato chips fried in the low linolenic acid soybean oil used for 10 h of frying. There were some differences between the composition of low linolenic acid soybean oil extracted from the potato chips compared to the fryer oil at the 20 h sampling time. These results showed that the supercritical carbon dioxide extraction gave similar results to hexane extraction in yield and composition of oils from potato chips.

Keywords: Supercritical fluid extraction, oil composition, tocopherols, cottonseed oil, low linolenic soybean oil, frying, fried food.

1 Introduction

There have been many studies involving frying of potato chips in various vegetable oils in order to observe the quality of the fried product [1-6]. Frying oils are exposed to extreme environmental conditions (air, water or steam, high temperatures of 140-200 °C), trace metals that result in degradation of the frying oil triacylglycerols by oxidation, polymerization, isomerization, cyclization and hydrolysis reactions [4-6]. These reactions affect the flavor quality of foods fried in the oils. Frying produces oils that are complex mixtures of unaltered triacylglycerols, triacylglycerols with conjugated diene and *trans* fatty acids, volatile compounds such as aldehydes, triacylglycerol oxidation products such as alkoxy, epoxy, keto monomeric compounds, higher molecular weight oxidation products, thermal degradation products such as oligomeric triacylglycerols or triacylglycerols with cyclized fatty acids and hydrolysis products such as diacylglycerols [6]. Additionally, frying oils, which have cooled to room temperature, contain triacylglycerol monohydroperoxides, that are only fleetingly present at frying oil temperatures [6]. A large number of the oxidation products have not been identified yet [6]. Many of the triacylglycerol oxidation products, which are formed, would be expected to be decomposed to volatile compounds during frying or heating [6]. While major quantities of the volatiles are steam-distilled out of the frying oil, some quantities of the volatiles remain in the oil and in the fried foods affecting flavor and odor of the food. To better control the production of undesirable flavor and odors, the respective volatile precursors or the molecular markers for the undesired volatile compounds need to be identified. Most reported studies of frying oil degradation products, which affect flavor, have involved various analyses of the fryer oil but not the oil absorbed by the fried food such as potato chips [4-6]. Very little work has been reported on the oil extracted from food products. Since the absorbed oil directly affects the flavor of the fried food, it is important to study the degradation products in this oil. Most extractions of lipids from foods use a labor-intensive and time-consuming process with large amounts of product extracted by hexane [7-10]. For analytical work, the hexane has to be removed; however, removal of hexane can produce artifacts, that can give misleading analytical results for degradation products. Also, hexane is an undesirable solvent and an environmentally unfriendly compound. Recently, supercritical fluid extraction has become more extensively utilized in the extraction of fats from foods using supercritical carbon dioxide, a nontoxic material [11-19]. Thus, a fat extract is obtained with little or no deterioration and with no solvent residue. How-

Correspondence: *Kathleen Warner*, National Center for Agricultural Utilization Research, Department of Agriculture, 1815 North University Street, Peoria, IL 61604, USA. Phone: +1-309-681-6584, Fax: +1-309-681-6668; e-mail: warnerk@ncaur.usda.gov

* Disclaimer: Names are necessary to report factually on available data; however, the United States Department of Agriculture (USDA) neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

Research Paper

ever, there exists no study comparing the efficacy of supercritical carbon dioxide extraction to the hexane-*Soxhlet* extraction referring to all the triacylglycerols, degradation products and non-triacylglycerol compounds such as tocopherols from a fried food product. Much current research on inherent antioxidants such as tocopherols require complete extraction of these materials from fried food to determine their retention levels. Therefore, our objective in this study was to compare compositions of oils extracted by supercritical carbon dioxide or hexane-*Soxhlet* from potato chips fried in cottonseed oil or low linolenic soybean oil.

2 Materials and methods

2.1 Materials

The oils used were commercially refined, bleached, and deodorized cottonseed oil (CSO) and low linolenic acid soybean oil (LLSBO). The oils contained only citric acid as an additive. Tocopherol samples were obtained from *Matreya*, Inc., Pleasant Gap, PA, USA. The standard of soybean oil oligomers (dimer, trimer, tetramer, *etc.*) was isolated from heated soybean oil [20]. The standard oleic series of triolein, diolein, monoolein and oleic acid was obtained from *NuChek* Prep, Inc. *Elysian*, MN, USA. Hexane, acetonitrile (ACN) and dichloromethane (DCM) used for extractions were HPLC grade. *Idaho Russet* variety potatoes were purchased locally.

2.2 Potato chip frying operation

The frying protocol included intermittent frying of potato chips at 190 °C with a total heating/frying time of 20 h. 800 g of each oil were heated in 1-l capacity fryers (*Presto Industries*, Model 2540, Eau Claire, WI, USA) for 6-7 h each day for 3 d. Fresh *Idaho Russet* potatoes were sliced, washed and fried in 100-g batches. Potato chip samples and fryer oil samples were taken at the 1, 10 and 20 h frying times. Each day 80 g of fresh oil were added as makeup oil to each fryer.

2.3 Supercritical carbon dioxide extraction

Supercritical fluid extractions (SFE) were performed with an ISCO Model 3560 SFE (ISCO Corporation, Lincoln, NE, USA). Potato chip samples (6.5 g) were mixed with ca. 2 g *Leco-Dry* (*Leco* Corporation, St. Joseph, MI, USA) and subsequently added to the extraction cell containing a glass fiber filter disk (18 mm diameter) on the bottom. Additional *Leco-Dry* was added to nearly fill the extraction cell and a second glass fiber filter was placed on top. The supercritical extractions were performed at 68.9 MPa and 70 °C at a flow rate of 2 ml/min for 45 min after an initial 1 min static hold. The variable restrictor was held at 55 °C and extracts were collected in 20 ml pre-cooled (5 °C) and

pressurized vials. 7 ml methylene chloride were used to clean the restrictor and were collected separately. The collection vials were purged under a gentle stream of nitrogen at room temperature and samples subsequently stored under nitrogen at –70 °C. SFE grade CO_2 (*Air Products and Chemicals* Inc., Allentown, PA, USA) was used for all extractions.

2.4 Hexane extraction

20 g of potato chips per extraction were extracted by hexane at 69 °C in a *Soxhlet* apparatus for 6 h.

2.5 Total polar compound analysis

The total polar compound analysis was conducted using the column chromatography methods from American Oil Chemists' Society Official Methods and Recommended Practices [21].

2.6 Tocopherol analysis

The α-, γ- and δ-tocopherol levels in the fresh oils, fryer oils, and oils extracted from potato chip samples were determined by a high-performance liquid chromatography (HPLC) with a polar phase column coupled with a fluorescence detector. The HPLC column used was a 3 micron particle size ultra silica HPLC column (25×0.49 cm) from *Phenomenex* (Torrance, CA, USA). The solvent system was 2% 2-propanol in hexane. The solvent was pumped at 0.5 ml/min. Sample size was 10 µl of 50 mg solute per ml of the mobile solvent. The fluorescence detector data was processed by the *Star* Workstation with version 4.0 software (*Varian*, Walnut Creek, CA, USA). The fluorescence detector was an HP programmable unit model 1046 A (with excitation wavelength set at 298 nm and emission wave length set at 345 nm with gain at 6) *Hewlett-Packard* Co (Palo Alto, CA, USA). The data output from the fluorescence detector was processed or integrated by a *Star* Chromatography Workstation. The tocopherol levels in the samples were expressed in chromatogram peak area counts. Linear standard curves of areas for α-, γ- and δ-tocopherol standards from 0.6-500 ppm concentrations were obtained. The tocopherol standards were prepared by appropriate dilutions of the standard tocopherol, 50 mg tocopherol, 99.4% pure, in 1 ml hexane.

2.7 Non-triacylglycerol analysis

Analysis of polymeric, monomeric, diacylglycerol, monoacylglycerol and free fatty acid components of the oils were obtained by size exclusion chromatography (SEC). SEC of the oil mixtures was performed on three, 30 x 7.5 cm, 5 µm particle size, PL-gel columns, PL Separation Sci-

ences, *Polymer Laboratories* Ltd. (Shropshire SY6 6AX, UK) in series. One column each of 50 nm, 10 nm and 5 nm (in this order) were used. DCM at 0.5 ml/min were used as the isocratic solvent for SEC. Twenty-five micron samples were injected in triplicate. The detector was an evaporative light-scattering detector (ELSD) *Sedex* Model 75, *Sedone* (Altontville, France). The drift tube was set at 32 °C. The gas-flow was set at a pressure of 1.6 hPa. The photomultiplier gain was times 4. High purity N_2 was used as the nebulizer gas. SEC chromatogram peak identification was in reference to a standard of soybean oil oligomers (dimer, trimer, tetramer, etc.) and to a standard oleic series of triolein, diolein, monoolein and oleic acid. The data output from the ELSD detector was processed or integrated by a *Star* Chromatography Workstation. The product levels in the samples were expressed in chromatogram peak area counts to give composition in area percent.

2.8 Triacylglycerol composition analysis

Reverse phase (RP)-HPLC was performed with a *Thermo Separation* Products (Schaumburg, IL, USA) (Model SP 8800) ternary solvent system with two RP-HPLC columns with bonded silyl (CT8) ODS, *Inertsil* ODS-80A, GL Sciences, *Keystone Scientific* (Bellefonte Park, PA, USA), 25 cm, 4.6 mm, 5 µm in series. The gradient elution was as follows: 80% acetonitrile (ACN), 20% dichloromethane (DCM) to 20% ACN, 80% DCM after 120 min. The flow rate was 0.5 ml/min. Sample size (25 mg) injected was 10 µl of 25 mg solute/ml DCM. Samples were injected in triplicate. The detector was an ELSD Sedex Model 75 operated as for SEC analysis. The data output from the ELSD detector was processed or integrated by a *Star* Chromatography Workstation. The product levels in the samples were expressed in chromatogram peak area counts to give triacylglycerol composition in area percent**.

2.9 Peroxide value determination

Peroxide values (PV) were determined in triplicate (15 mg samples) by the previously reported colorimetric ferric thiocyanate method [22].

2.10 Statistical analyses

Statistical analyses of three replicate analyses of α-, γ- and δ-tocopherols were conducted to determine effects of oil source and frying time. Analyses of variance (ANOVA) were performed with *Statistix* 7 software (*Analytical Software*, Tallahassee, FL, USA). Means were compared using least significant difference (LSD) at the P = 0.05 level.

3 Results and discussion

3.1 Hexane and supercritical fluid extraction

The objectives of this study included methodology development for SFE to potentially replace hexane extraction of oil absorbed by potato chips during frying. The SFE method used a much smaller sample (2.5 g) than the hexane extracted potato chip sample, which required 20 g for the 6 h *Soxhlet* extraction. The SFE procedure used a much shorter extraction time (45 min) compared to the 6 h hexane procedure. In addition, no solvent had to be removed from extract obtained by SFE. Shorter extraction times and no solvent removal probably reduced artifact formation in the SFE samples compared to the hexane-extracted potato chips. Finally, SFE is safer and environmentally less hazardous than hexane extraction.

3.2 Oil recovery and decomposition products

In the development and validation of the SFE procedure, it was important to determine any differences in composition and yield in oil extracted from the potato chips. For cottonseed oil, overall mean ($\pm$ standard deviation) oil recoveries for SFE and hexane extractions were 47.5% ($\pm$1.9%) and 49.0% ($\pm$2%), respectively. For low linolenic acid soybean oil, overall mean oil recoveries were 46.5% ($\pm$1.9%) and 48.0% ($\pm$2%), respectively. The SFE procedure was compared with the hexane extraction procedure for potato chips fried 20 h in cottonseed oil. Data for the 6 h hexane extraction of 25.98 g of chips showed 47.0% oil yield with a PV of 8.33 meq peroxide/kg and 17.29% total polar compounds. Data from the 1 h SFE of 5.70 g chips showed 45.6% oil yield with PV of 9.02 meq peroxide/kg and 17.54% total polar compounds. The differences in recovery of oil between SFE and hexane extrac-

Tab. 1. Mean (n = 3) concentrations (ppm) of α-and γ-tocopherols in fryer oils and in potato chips extracted from cottonseed oil after 1, 10 and 20 h of frying by either supercritical fluid extraction (SFE) or by hexane[†].

Oil source	Alpha			Gamma		
	1	10	20	1	10	20
Fryer oil	690[a]	470[a]	476[a]	233[a]	150[a]	126[a]
SFE	606[a]	520[a]	493[a]	213[a]	163[a]	146[a]
Hexane	720[a]	563[a]	546[a]	240[a]	193[a]	176[a]

[†] Means within each column with letters in common are not significantly different (P $\geq$ 0.05).

Tab. 2. Mean (n = 3) concentrations (ppm) of γ- and δ-tocopherol in fryer oils and in potato chips extracted by either supercritical fluid extraction (SFE) or by hexane from low linolenic soybean oil at 1, 10 and 20 h of frying[†].

Oil Source	Gamma			Delta		
	1	10	20	1	10	20
Fryer oil	460[a]	313[a]	160[a]	160[a]	140[a]	53[a]
SFE	440[a]	323[a]	230[b]	150[a]	146[a]	116[b]
Hexane	436[a]	250[a]	223[b]	160[a]	77[b]	100[b]

[†] Means within each column with letters in common are not significantly different (P ≥ 0.05).

Tab. 3. Triacylglycerol and non-triacylglycerol compositions of cottonseed oil[†].

Analyses	0 time Oil source	1 h Fryer oil	Hexane	SFE	10 h Fryer oil	Hexane	SFE	20 h Fryer oil	Hexane	SFE
Oligomer	0	0	0	0	0	0	0	0	0	0
Trimer	0	0	0	0	0	0	0	0	0	0
Dimer	0.1	0.5	0.3	0.3	0.4	0.9	0.8	0.9	1.3	1.8
Monomer	99.7	99.1	99.3	99.4	99.4	99	98.9	99	98.5	97.7
DAG	0.3	0.5	0.4	0.4	0.2	0.2	0.3	0.1	0.2	0.5
MAG	0	0	0	0	0	0	0	0	0	0
FFA	0	0	0	0	0	0	0	0	0	0
Total TAG	99.7	99.1	99.3	99.4	99.4	99	98.9	99	98.5	97.7
Total NonTAG	0.3	0.9	0.7	0.6	0.6	1	1.1	1	1.5	2.3
TAG										
LnLnLn	0	0	0	0	0	0	0	0	0	0
LnLnL	0	0	0	0	0	0.1	0.1	0.1	0	0.1
LnLL	0	0	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
LnLnO	0	0	0	0	0	0.1	0.1	0.1	0.1	0.1
LnLnP	0	0	0	0	0	0.1	0.1	0.1	0	0
LLL	16.4	16.7	16.9	16.8	15	14.5	14.6	14.2	14.8	13.8
LnLO	0.1	0.4	0.4	0.4	0.5	0.6	0.5	0.6	0.5	0.4
LnLP	0.4	0.4	0.4	0.4	0.5	0.7	0.6	0.6	0.5	0.4
LLO	10.9	10.8	10.8	10.6	11.2	10.6	11	10.4	11	10.2
LnOO	0	0.1	0.1	0.1	0.2	0.2	0.2	0.2	0.2	0.2
LLP	33.5	32.2	34.7	33.9	28.6	27.9	28.5	28	29.5	30.1
LnOP	0.7	0.7	0.7	0.7	1	1.2	1	1.1	0.9	0.9
LnPP	0.4	0.4	0.4	0.3	0.1	0.1	0.7	0.1	0.1	0.5
LOO	2.5	2.6	2.4	2.4	3.9	3.8	3.2	3.7	3.6	2.8
LLS	1.1	1.1	1	1	1.5	1.5	1.5	1.4	1.4	1.3
LOP	14.4	14.5	13.6	13.9	14.5	14.4	14.8	14.7	14.6	15.3
PLP	13.3	13.7	12.6	13.2	13.7	13.9	14	14.4	13.8	15.2
OOO	0.7	0.6	0.6	0.6	1	1.1	1	1.1	1	0.9
LOS	0.5	0.4	0.4	0.4	0.7	0.7	0.7	0.6	0.6	0.5
POO	1.8	1.7	1.6	1.7	2.3	2.5	2.4	2.5	2.3	2.3
SLP	0.9	0.9	0.8	0.9	1.2	1.4	1.3	1.4	1.2	1.2
POP	2.4	2.4	2.2	2.3	3.2	3.5	3.2	3.6	3.1	3.4
PPP	0	0	0	0	0	0	0	0	0	0
SOO	0	0.1	0.1	0.1	0.2	0.3	0.3	0.3	0.2	0.3
SLS	0	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
SOP	0.2	0.2	0.2	0.2	0.3	0.3	0.3	0.4	0.3	0.3
PPS	0	0	0	0	0.1	0.1	0.1	0.1	0.1	0.1
SOS	0	0	0	0	0.1	0.1	0.1	0.1	0.1	0.1
PSS	0	0	0	0	0	0.1	0.1	0.1	0	0.1
SSS	0	0	0	0	0	0	0	0	0	0.1

[†] See experimental section for analytical methods.

Tab. 4. Triacylglycerol and non-triacylglycerol composition of low linolenic acid soybean oil.

Analyses	0 time Oil source	1 h Fryer oil	Hexane	SFE	10 h Fryer oil	Hexane	SFE	20 h Fryer oil	Hexane	SFE
Oligomer	0	0	0	0	0	0	0	0	0	0
Trimer	0	0	0	0	0	0	0	0	0	0
Dimer	0	0.1	0.1	0.2	0.7	1.3	1.1	0.7	1.7	2
Monomer	99.9	99.8	99.7	99.6	99.2	98.6	98.7	99.2	98.1	97.8
DAG	0.1	0.2	0.1	0.2	0.1	0.1	0.2	0.1	0.2	0.2
MAG	0	0	0	0	0	0	0	0	0	0
FFA	0	0	0	0	0	0	0	0	0	0
Total TAG	99.9	99.8	99.7	99.6	99.2	98.6	98.7	99.2	98.1	97.8
Total NonTAG	0.1	0.2	0.3	0.4	0.8	1.4	1.3	0.8	1.9	2.2
TAG										
LnLnLn	0	0	0	0	0	0	0	0	0	0
LnLnL	0	0.1	0.1	0	0.1	0	0.1	0.1	0	0.1
LnLL	1.6	1.5	1.6	1.7	1.3	0.9	1.3	1.1	1.1	1.2
LnLnO	0.1	0.1	0.1	0.1	0.2	0.1	0.2	0.2	0.1	0.3
LnLnP	0	0	0	0	0	0	0	0	0	0
LLL	23.5	22.6	21.6	19.9	21.5	23.6	21.8	20.7	23.7	19.1
LnLO	1.7	1.7	1.8	1.9	1.7	1.2	1.7	1.6	1.5	1.8
LnLP	0.6	0.6	0.7	0.8	0.6	0.3	0.6	0.6	0.4	0.7
LLO	21.8	21.9	21.5	20.5	21.7	23.7	21.9	21.7	23.2	20.5
LnOO	0.5	0.5	0.6	0.6	0.5	0.4	0.6	0.5	0.4	0.7
LLP	14.5	13.9	13.7	14.1	14	14.6	14	13.7	14.4	13.6
LnOP	0.3	0.4	0.4	0.5	0.4	0.3	0.4	0.4	0.3	0.5
LnPP	0.1	0.1	0.1	0.2	0.1	0.1	0.1	0.1	0.1	0.1
LOO	10.6	10.5	10.7	11.2	10.9	10.8	10.9	11.2	10.9	11.2
LLS	3.7	3.8	4	4.2	3.7	3.2	3.7	3.8	3.3	3.9
LOP	9.4	9.4	9.4	9.8	9.7	9.6	9.8	10.1	9.7	10.1
PLP	1.6	1.5	1.7	1.9	1.6	1.3	1.7	1.7	1.3	2.1
OOO	3	3.4	3.5	3.7	3.5	3.1	3.4	3.7	3.1	4
LOS	2.6	2.8	3	3.1	2.8	2.5	2.8	2.9	2.4	3.2
POO	2.2	2.3	2.4	2.6	2.5	2.2	2.5	2.6	2.1	3
SLP	0.8	0.9	1	1.1	1	0.7	1	1	0.7	1.2
POP	0.4	0.5	0.6	0.6	0.6	0.4	0.6	0.6	0.4	0.7
PPP	0	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
SOO	0.6	0.7	0.7	0.8	0.8	0.6	0.8	0.8	0.6	1
SLS	0.1	0.1	0.2	0.2	0.2	0.1	0.2	0.2	0.1	0.3
SOP	0.2	0.3	0.3	0.4	0.3	0.2	0.3	0.4	0.3	0.5
PPS	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
SOS	0	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.2
PSS	0	0	0	0	0.1	0.1	0.1	0.1	0.1	0.1
SSS	0	0	0	0	0	0	0	0	0	0

tion procedures for potato chips were not significant. The closeness of the peroxide value and polar component percent of the extracted oils showed that both procedures extracted the same amount of degradation products and the same amount of unreacted triacylglycerol.

3.3 Oil composition

To determine the composition of the fryer oils and the oils extracted by SFE and hexane, tocopherol, triacylglycerol, polymer, monomer, diacylglycerol, monoacylglycerol, and free fatty acids were measured. Knowledge of tocopherol concentration in the oil absorbed by potato chips and in the fryer oil can be used to study the pro-oxidant or antioxidant effects of minor constituents in oils and foods. The ANOVA for α-tocopherol levels in cottonseed fryer oil and potato chips fried in cottonseed oil indicated that there was no significant effect of oil source (hexane or SFE extraction or fryer oil) (P = 0.21) (Tab. 1). The interaction was not significant (P = 0.77). As expected, time was significant (P = 0.001) because α-tocopherol de-

creased with increasing frying time. Similarly, γ-tocopherol levels in cottonseed oil and chips showed no significant difference from of oil source (P = 0.09), but frying time was significant (P = 0.001). The interaction was not significant (P = 0.76). For cottonseed oil, γ-tocopherol concentrations also decreased as frying time increased (Tab. 1).

γ-Tocopherol levels in low linolenic soybean oil indicated no significant effect of extraction type at 1 and 10 h of frying (P = 0.07) (Tab. 2). However, at the 20 h frying time, there was a significant difference between the γ-tocopherol level in the fryer oil and the amount of γ-tocopherol in either of the extracted oils (P = 0.05). Frying time was significant (P = 0.0001), as γ-tocopherol in low linolenic acid soybean oil decreased as frying time increased (Tab. 2). The interaction was significant too (P = 0.002). The ANOVA for the δ-tocopherol in low linolenic soybean oil showed no significant difference between oil sources at the 1 h frying time (P = 0.07). At the 10 h time, we found the only instance in this study of a significant difference related to the extraction method, as the oil extracted with SFE contained significantly more δ-tocopherol than the hexane extracted oil (P = 0.02). At the 20 h frying time, both extracted oils had significantly more δ-tocopherol than the fryer oil sample (P = 0.04), which was the same result found for γ-tocopherol in the 20 h samples. Significant effects of frying time (P = 0.0001) as well as a significant interaction (P = 0.001) were observed as well. δ-Tocopherol concentrations in low linolenic soybean oil tended to decrease as frying time increased (Tab. 2).

Non-triacylglycerol compositions presented in Tab. 3 for cottonseed oil and in Tab. 4 for low linolenic acid soybean oil showed no differences between the SFE and hexane extraction of degradation products with fry time. There were few differences between the non-triacylglycerol composition of the extracted potato chip oil and fryer oil with frying time. Triacylglycerol compositions presented in Tab. 3 for cottonseed oil and in Tab. 4 for low linolenic acid soybean oil showed little difference between the SFE and hexane procedures for extraction of triacylglycerols with frying time. Also, there was little difference between the triacylglycerol composition of the extracted potato chip oils and fryer oils at all frying times. An increase of saturated fatty acids in triacylglycerol was observed compared to a decrease in triacylglycerol with unsaturated fatty acids with increased frying times for the fryer and extracted oils.

SFE and hexane extraction of oils from potato chips produced similar tocopherol compositions at each fry time with the exception of the δ-tocopherol levels in the 10 h low linolenic acid soybean oil sample. Differences were noted between tocopherol composition of the fryer oil and the extracted oil for the 20 h low linolenic acid soybean oil

sample. The SFE and hexane extraction and fryer oils had similar compositions of unreacted triacylglycerol as well as thermal, oxidative and hydrolytic degradation products at each fry time. Similar decreases in levels of α- and γ-tocopherols and increases in thermal and hydrolytic products were observed in both cottonseed oil and low linolenic acid soybean oil as frying time increased. Oil recoveries from both procedures were comparable. These results showed that SFE produced oils that had the similar composition as oils obtained by the more labor-intensive hexane extraction procedure. Also oils extracted by SFE and hexane had the similar triacylglycerol and degradation product composition as the fryer oils. In cottonseed and low linolenic acid soybean oils, the same increase in triacylglycerol with saturated fatty acids and decrease in triacylglycerol with unsaturated fatty acids with fry time was observed for the extracted and fryer oils.

References

[1] *K. Warner, T. L. Mounts:* Frying stability of soybean and canola oils with modified fatty acid compositions. J. Am. Oil Chem. Soc. **70** (1993) 983-988.

[2] *K. Warner, P. Orr, L. Parrott, M. Glynn:* Effects of frying oil composition on potato chip stability. J. Am. Oil Chem. Soc. **71** (1994) 1117-1121.

[3] *K. Warner, P. Orr , P. Glynn:* Effect of fatty acid composition of oils on flavor and stability of fried foods. J. Am. Oil Chem. Soc. **74** (1997) 347-356.

[4] *C. Getz:* Chemical changes of oils and fats at elevated temperatures. PJ Barnes and Associates, Bridgwater (England) 1995, pp. 577-582.

[5] *K. Warner:* Chemistry of frying fats. In: food chemistry, nutrition, and biotechnology. Eds. *C. C. Akoh, D. B. Min*, Marcel Dekker, New York (USA) 1998, pp.167-180.

[6] *E. N. Frankel:* Lipid oxidation. The Oily Press, Dundee (Scotland) 1998, 227-248.

[7] *G. Granata, R. H. Lane*: Crude oil content of selected oilseeds and flours: a comparison of solvents. J. Assoc. Off. Anal. Chem. **74** (1991) 692-694.

[8] *L. Di Giovacchino, M. Solinas, M. Miccoli*: Effect of extraction systems on the quality of virgin olive oil. J. Am. Oil Chem. Soc. **71** (1994) 1189-1194.

[9] *A. Koutsaftakis, F. Kotsifaki, E. Stefanoudaki*: Effect of extraction system, stage of ripeness, and kneading temperature on the sterol composition of virgin olive oils. J. Am.Oil Chem. Soc. **76** (1999) 1477-1481.

[10] *A. Ranalli, M. L. Ferrante, G. De Mattia, N. Costantini*: Analytical evaluation of virgin olive oil of first and second extraction. J. Agric. Food Chem. **47** (1999) 417-424.

[11] *D. J. Charles, J. E. Simon:* Comparison of extraction methods for the rapid determination of essential oil content and composition of basil. J. Amer. Soc. Hort. Sci. **115** (1990) 458-462.

[12] *M. E. Ramsay, J. T. Hsu, R. A. Novak, W. J. Reightler*: Processing rice bran by supercritical fluid extraction. Food Tech. **45** (1991) 98-104.

[13] *E. Reverchon, F. Senatore*: Supercritical carbon dioxide extraction of chamomile essential oil and its analysis by Gas Chromatography-Mass Spectrometry. J. Agric. Food Chem. **42** (1994) 154-158.

[14] *N. O. Maness, D. Chrz, T. Pierce, G. H. Brusewitz*: Quantitative extraction of pecan oil from small samples with supercritical carbon dioxide. J. Am. Oil Chem. Soc. **72** (1995) 665-669.

[15] *B. Simandi, M. Oszagyan:* Comparison of the volatile composition of chervil oil obtained by hydrodistillation and supercritical fluid extraction. J. Essent. Oil Res. **8** (1996) 305-306.

[16] *M. Oszagyan, B. Simandi, J. Sawinsky:* A comparison between the oil and supercritical carbon dioxide extract of hungarian wild thyme (*Thymus serpyllum* L.). J. Essent. Oil Res. **8** (1996) 333-335.

[17] *S. L. Taylor, F. J. Eller, J. W. King*: A comparison of oil and fat content in oilseeds and ground beef—using supercritical fluid extraction and related analytical techniques. Food Res. Int. **30** (1997) 365-370.

[18] *J. A. Pino, J. Garcia, M. A. Martinez:* A comparison between the oil, solvent extract and supercritical carbon dioxide extract of *Ocimum gratissimum* L. J. Essent. Oil Res. **10** (1998) 575-577.

[19] *E. Ibanez, A. Oca, G. de Murga, S. Lopez-Sebastian, J. Tabera, G. Reglero*: Supercritical fluid extraction and fractionation of different preprocessed rosemary plants. J. Agric. Food Chem. **47** (1999) 1400-1404.

[20] *W. E. Neff, K. Warner, W. C. Byrdwell:* Odor significance of undesirable degradation compounds in heated triolein and trilinolein. J. Am. Oil Chem. Soc. **77** (2000) 1303-1313.

[21] American Oil Chemists' Society Official Methods and Recommended Practices. 5th ed., American Oil Chemists' Society, Champaign, IL (USA), 1998.

[22] *W. E. Neff, T. L. Mounts, W. M. Rinsch, H. Konishi, M. A. El-Agaimy:* Oxidative stability of purified canola oil triacylglycerols with altered fatty acid composition as affected by triacylglycerol composition and structure. J. Am. Oil Chem. Soc. **71** (1994) 1101-1109.